REPORT NO. 11

RESEARCH AND DEVELOPMENT PROGRAM INTRINSIC RELIABILITY SUBMINIATURE CERAMIC CAPACITORS

ELEVENTH QUARTERLY PROGRESS REPORT

PERIOD: 1 DECEMBER 1964 - 28 FEBRUARY 1965

TO

U. S. ARMY SIGNAL RESEARCH & DEVELOPMENT LABORATORY
FORT MONMOUTH, NEW JERSEY

CONTRACT NO. DA-36-039-SC-90705
D. A. PROJECT NO. 1P6 22001 A 057

SPRAGUE ELECTRIC COMPANY
NORTH ADAMS, MASSACHUSETTS

463306

Report No. 11

RESEARCH AND DEVELOPMENT PROGRAM

INTRINSIC RELIABILITY

SUBMINIATURE CERAMIC CAPACITORS

Eleventh Quarterly Report

Period: 1 December 1964 - 28 February 1965

Object of Study: To conduct investigations leading to the approaches for the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

Contract No. DA-36-039-SC-90705
D. A. Project No. 1P6 22001 A 057

Controlling Specifications:

Signal Corps Technical Guidelines, "Reliability Long Life Component Studies," 3 November 1961
Signal Corps Technical Requirements No. SCL-2101N, 14 July 1961

Report Prepared by:

P. M. Kennedy
T. I. Prokopowicz

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SECTION 1

PURPOSE

The purpose of this contract is to carry out research work involving investigations leading to approaches to the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

In particular, this involves the following:

(1) Construction of a model or theory to predict failure mechanisms and failure rates as a function of voltage and temperature.

(2) Development and evaluation of a short-term test to eliminate early failures effectively without shortening the time to the wearout mode of failure.

(3) A determination of the failure rate as a function of voltage and temperature through large-scale life testing. From the data thus obtained, derating curves will be derived and overall failure rates for operating conditions will be estimated. The theory developed will be critically examined and refinements made.

(4) Compilation of quarterly progress reports in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.

(5) Compilation of a final report in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.

SECTION 2

ABSTRACT

Testing of C67 Case Size I MONOLYTHIC® capacitors of the improved version continues to demonstrate the long-life capability of this capacitor at use conditions. The testing of 124 of these capacitors at 200 VDC and 125°C for 2500 hr without catastrophic failure, then at 400 VDC and 125°C for 4000 hr with only five displaying any degradation, continues to indicate the intrinsic high reliability of this unit.

Some properties of 1800 capacitors (0.01 μf rating) manufactured for voltage-temperature matrix testing were examined.

The duration of the contract has been extended to permit statistical demonstration of the validity of the long life capacitor selection technique.

SECTION 3

PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

The Tenth Quarterly Progress Report, covering the period September 1, 1964 - November 30, 1964, was submitted for U. S. Army Electronics Research and Development Agency approval.

SECTION 4

FACTUAL DATA

4.1 Construction of C67 Case Size I MONOLYTHIC Capacitors

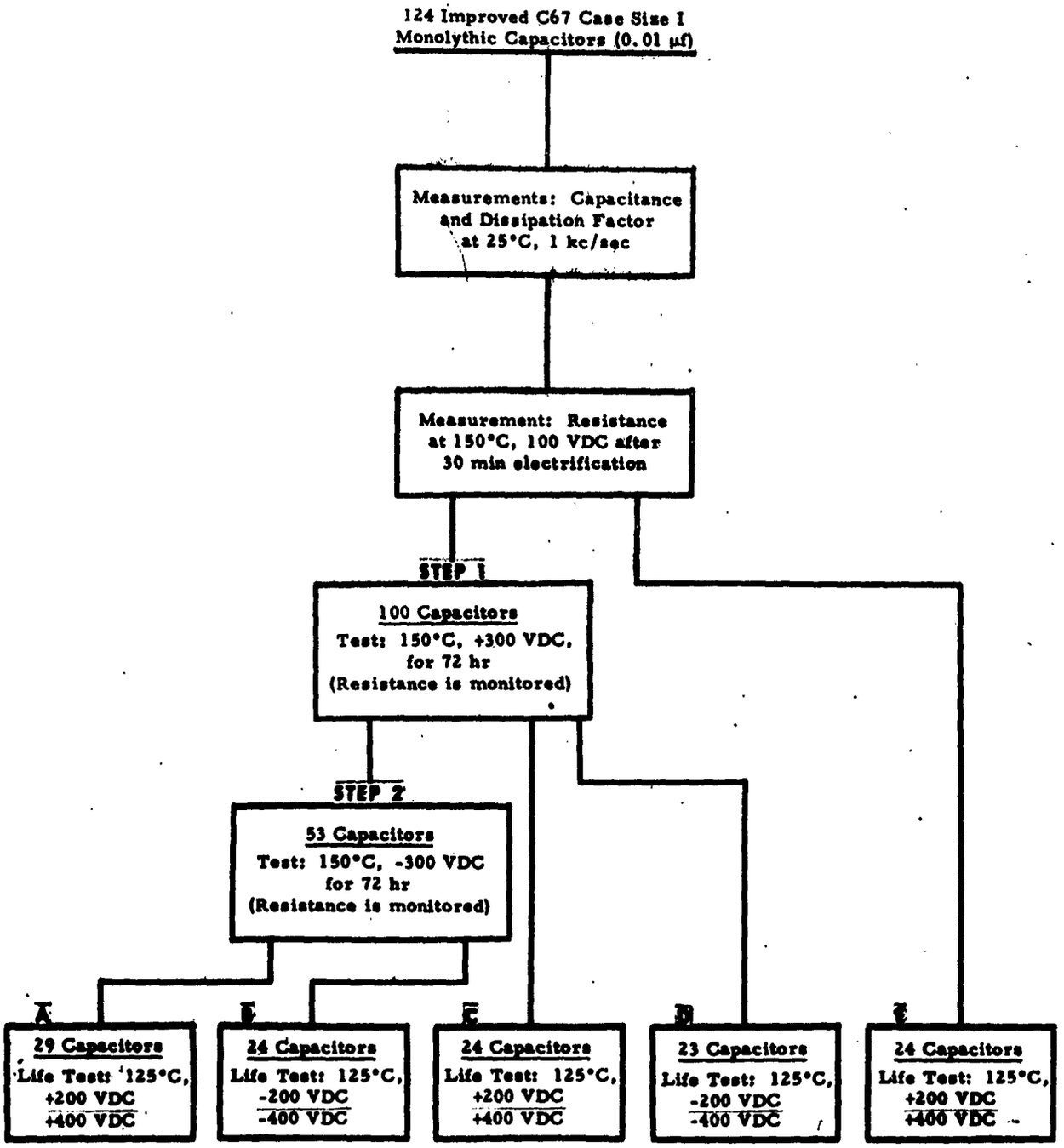
The C67 Case Size I MONOLYTHIC capacitor has a construction of stacked ceramic dielectric layers 0.0025 in. thick and connected electrically in parallel. These layers are intimately bonded to each other through high-temperature sintering. The ceramic material is a barium titanate, which has a dielectric constant of about 2000 at room temperature and is stable to +10%, -15% between -55°C and +125°C. The capacitor is enclosed in a tubular case which is 0.25 in. long and has a diameter of 0.095 in.

4.2 Selection of Long Life Capacitors of the Improved Version

An experiment designed to establish a procedure for the selection of long life capacitors of the improved version was begun during the eighth quarter and is still in progress. An outline of the various parts of the experiment is presented in Figure 1. Basically, the plan was to demonstrate that life lost by a capacitor during a brief period of accelerated testing can be regained by application of direct voltage of opposite sense for some given time. Furthermore, it was planned to demonstrate that if a particular capacitor is capable of a given performance at some accelerated condition, it will deliver at least a certain minimum standard of performance at milder conditions. This latter objective was obtained during the ninth quarter and was reported in the Ninth Quarterly Progress Report.

Properties of the 124 C67 Case Size I MONOLYTHIC capacitors of the improved version chosen for the experiment were described in the Eighth Quarterly Progress Report. Of these 124 capacitors, 53 were subjected to a two-part screening procedure incorporated into the experiment outlined in Figure 1. The screening procedure comprised the following steps:

Step 1 - The capacitors are subjected to 300 VDC at 150°C for 72 hr, during which time the resistance of acceptable capacitors may not vary more than 20%.



EXPERIMENT TO SELECT CAPACITORS HAVING POTENTIALLY LONG LIVES

Figure 1

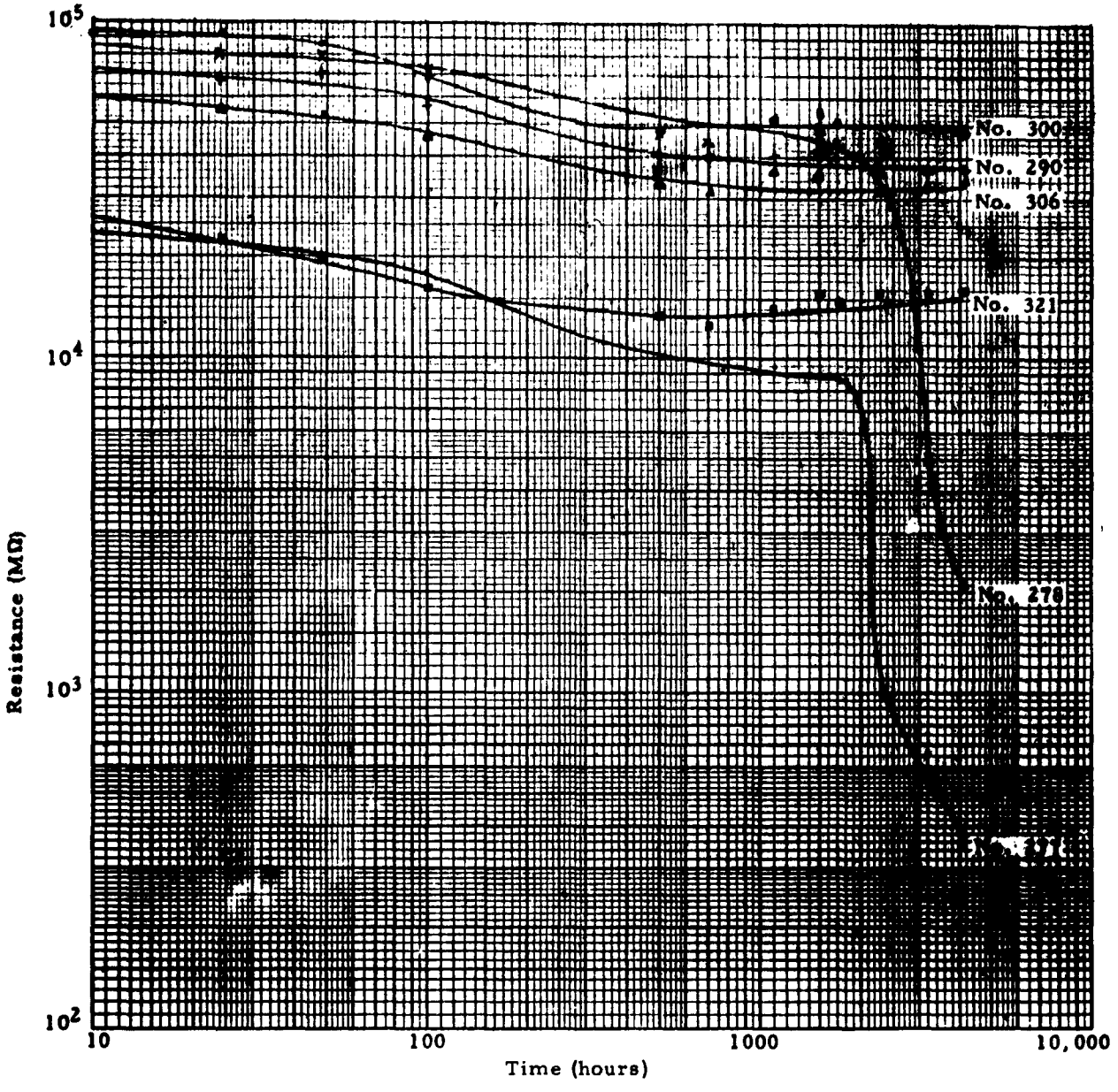
Step 2 - The acceptable capacitors from Step 1 are subjected to 300 VDC, applied in the opposite sense at 150°C for 72 hr, during which time the resistance of acceptable capacitors may not vary more than 20%.

The purpose of Step 1 of the screening program is to identify those capacitors which have not reached the point of onset of degradation. The purpose of Step 2 is to rejuvenate normal capacitors and to detect those capacitors which are sensitive to voltage polarity because of a construction or material defect.

Life testing of the 124 capacitors was conducted at 125°C and 200 VDC for 2500 hr. None of the 53 capacitors which passed the screening procedure had reached the onset of degradation by the end of that time. (These capacitors are represented in Positions A and B of Figure 1.) Information regarding this life testing was published in the Ninth Quarterly Progress Report.

At the end of 2500 hr of testing at 125°C and 200 VDC, the life test voltage on the 124 capacitors was increased to 400 VDC. A total of 4000 hr of test time have been accumulated at this new condition. Two of the 53 capacitors which were subjected to both steps of the screening procedure are displaying marked degradation. Figure 2 shows resistance plotted as a function of time for several of the 53 capacitors. The two non-typical capacitors, Nos. 277 and 278, had initial RC products of 63 MΩ-μf and 200 MΩ-μf, respectively, at 150°C. This should be compared with the average RC product value of 130 MΩ-μf shown in Figure 18 of the Eighth Quarterly Progress Report. The relative resistances these two capacitors exhibited as a consequence of Step 1 in the screening procedure were 0.99 and 0.96, respectively. This should be compared with the average value of 0.94 shown in Figure 20 of the Eighth Quarterly Progress Report. The relative resistances these two non-typical capacitors exhibited as a consequence of Step 2 in the screening procedures were 0.90 and 0.88, respectively. This may be compared with the average value of 0.91 indicated in Figure 23 of the Eighth Quarterly Progress Report. It may be concluded that none of the measured values for Capacitor Nos. 277 and 278 before life test can be suspected to indicate the non-typical behavior noted for these capacitors during life test at 400 VDC, 125°C.

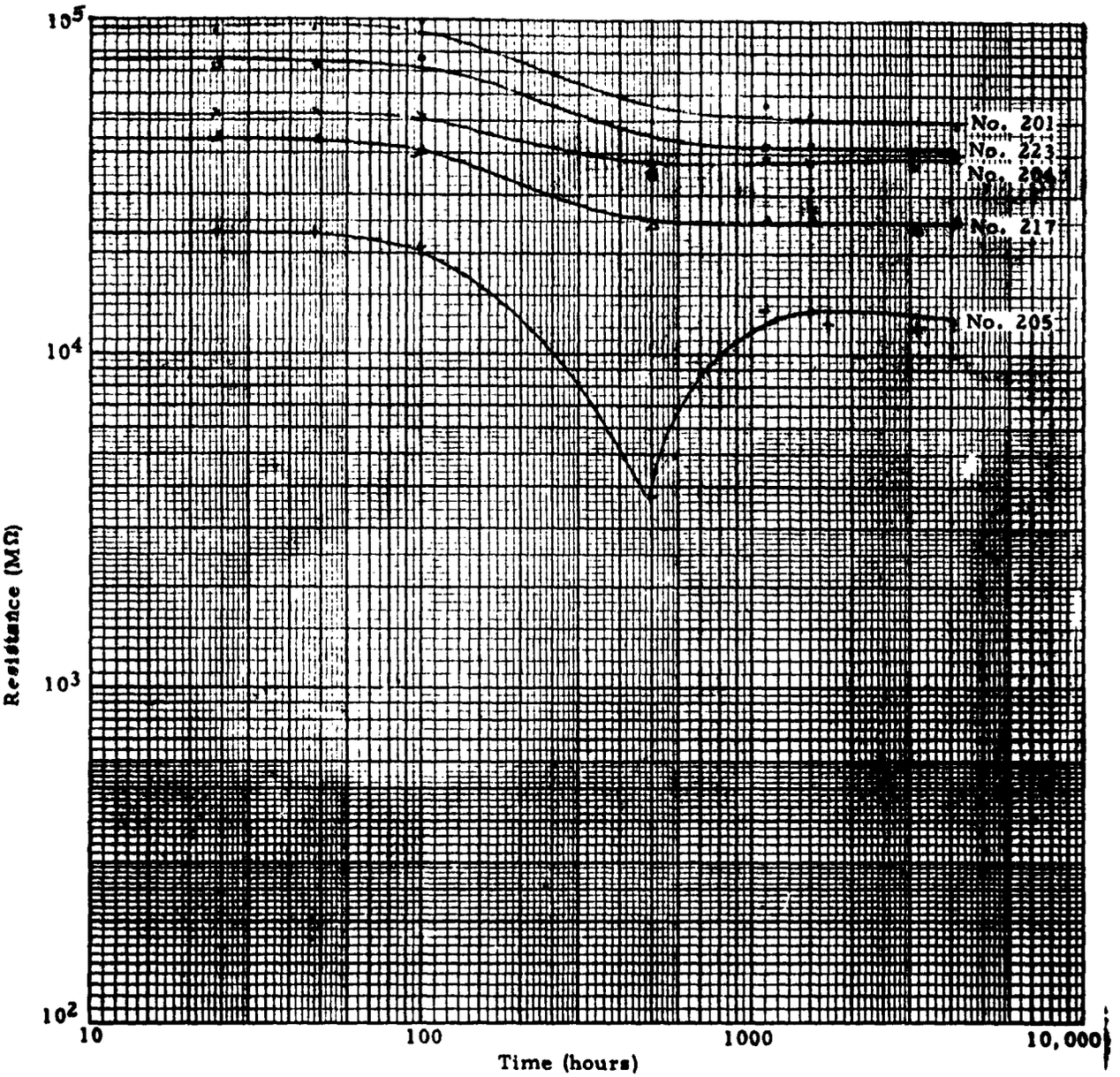
Resistance as a function of time for capacitors which were not subjected to either screening step is shown in Figure 3. These capacitors are represented in Position E in Figure 1. The general behavior of capacitors subjected to the screening procedure is similar to these. The slight decrease in resistance between 100 hr and 500 hr is due to a slight



RESISTANCE VS TIME AT 125°C, 400 VDC
FOR 0.01 μ E SIZE I C67 MONOLYTHIC CAPACITORS (Lot 830)

(Capacitors were previously at 125°C, 200 VDC for 2500 hours and were subjected initially to both steps of screening procedure)

Figure 2



RESISTANCE VS TIME AT 125°C, 400 VDC
FOR 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 830)

(Capacitors were previously at 125°C, 200 VDC for 2500 hours)

Figure 3

increase in temperature in the test oven from 124°C to 127°C. There is no explanation for the behavior of Capacitor No. 205, whose RC product at 150°C, 100 V before life testing was 67 MΩ-μf. This should be compared with the average RC product value of 130 MΩ-μf presented in Figure 18 of the Eighth Quarterly Progress Report.

The behavior of resistance with time for several capacitors which were subjected to Step 1, but not Step 2, of the screening procedure is shown in Figure 4. These capacitors are represented in Positions C and D in Figure 1. Three capacitors, Nos. 246, 253, and 260, were found unacceptable when subjected to Step 1 of the screening procedure. Their behavior in Step 1 was reported in Figure 21 of the Eighth Quarterly Progress Report.

The testing of the 124 capacitors is continuing.

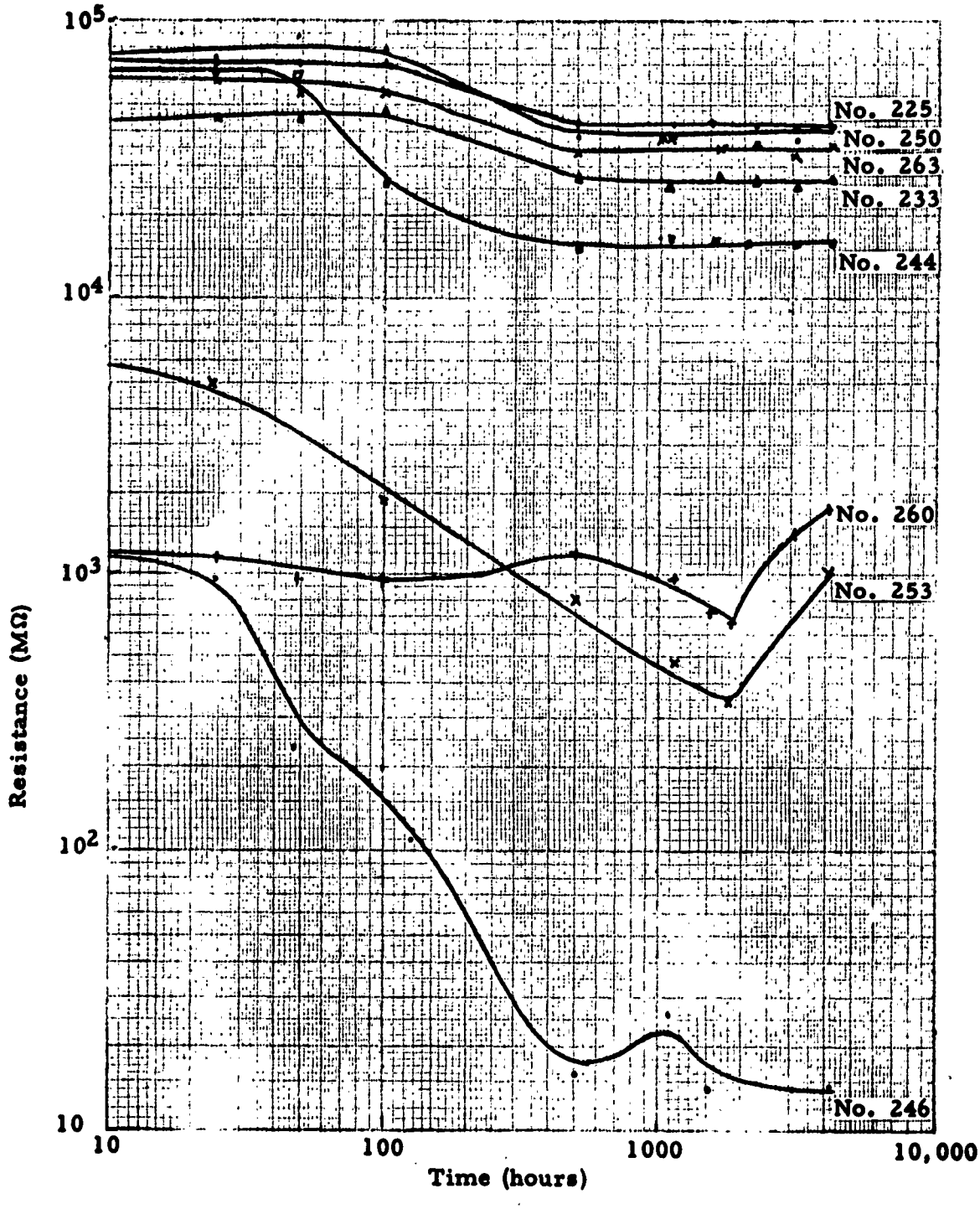
4.3 Voltage-Temperature Matrix

Delivery of 1800 capacitors (0.01 μf) of the improved version to the Sprague test laboratory was completed toward the end of the eleventh quarter. A total of 1500 capacitors will be tested for 10,000 hr. Before placing the capacitors on life test they will be screened, and only potentially long life capacitors will be selected for testing. The number of capacitors to be subjected to the various test conditions is shown in Table 1.

Among the objectives sought in performing the voltage-temperature matrix testing are the statistical derivation of formulas relating lifetime to both these parameters, the statistical evaluation of the screening technique for selecting long-life capacitors, and the determination of the relationship of temperature and voltage to the degradation rate.

Before subjecting the 1800 capacitors (Lot No. 6S9205) to the screening procedure described earlier in this report and then to voltage-temperature matrix testing, it seemed advisable to examine, on a sample basis, the properties of these capacitors and to learn to what degree the properties differ from those of capacitors examined previously.

Figure 5 shows current charging curves at 150°C and 195 V for several of the 1800 capacitors of Lot No. 6S9205 delivered for voltage-temperature matrix testing; Figure 6 presents discharge current curves for the same capacitors. (The equipment and method for measuring charge and discharge current were described in the Fourth Quarterly Progress Report.) Figures 7 and 8 present, respectively, the current charging and discharging curves at 25°C.



RESISTANCE VS TIME AT 125°C, 400 VDC
FOR 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 830)

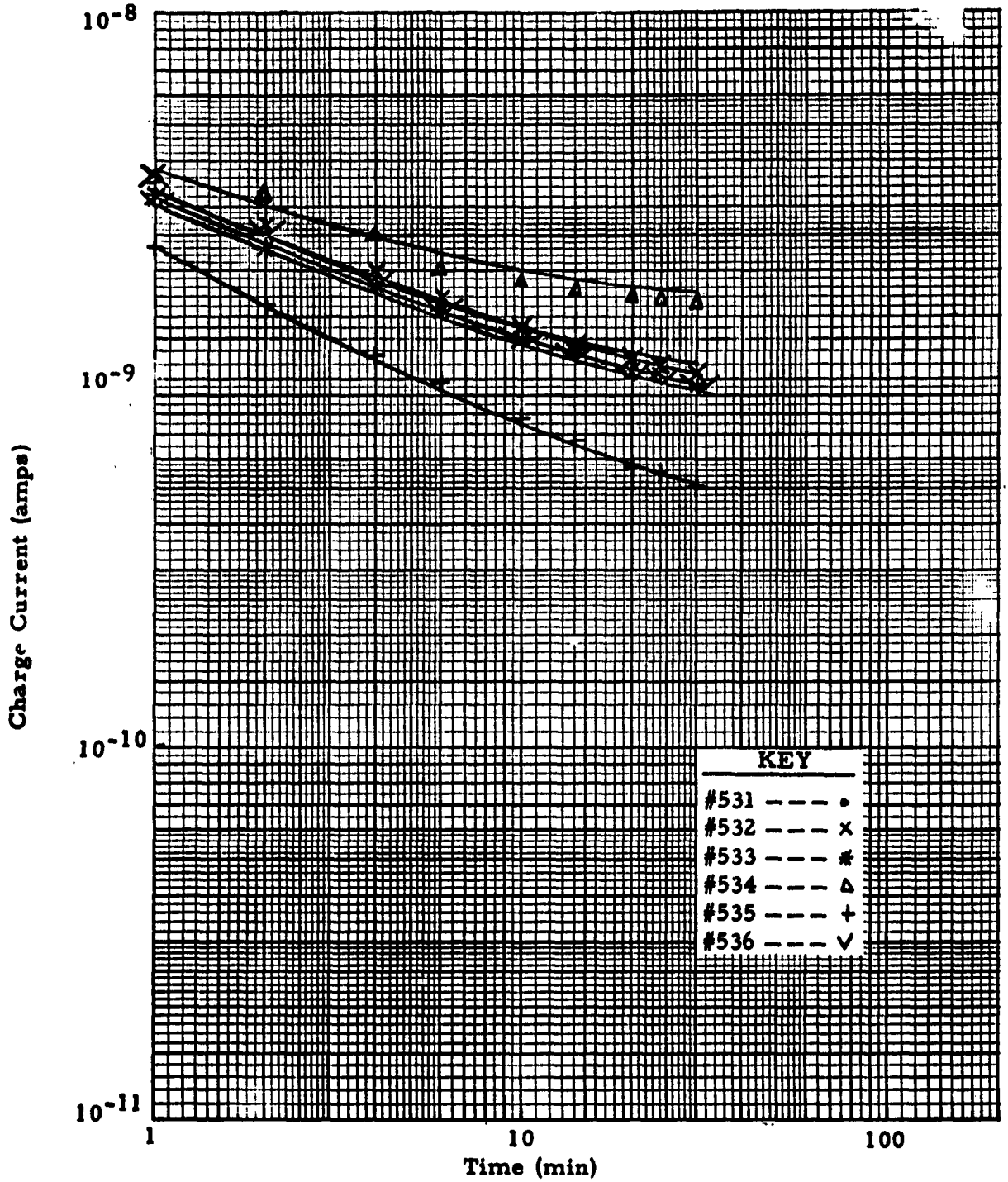
(Capacitors were previously at 125°C, 200 VDC for 2500 hours and were subjected initially to Step 1 of screening procedure)

Figure 4

TABLE 1

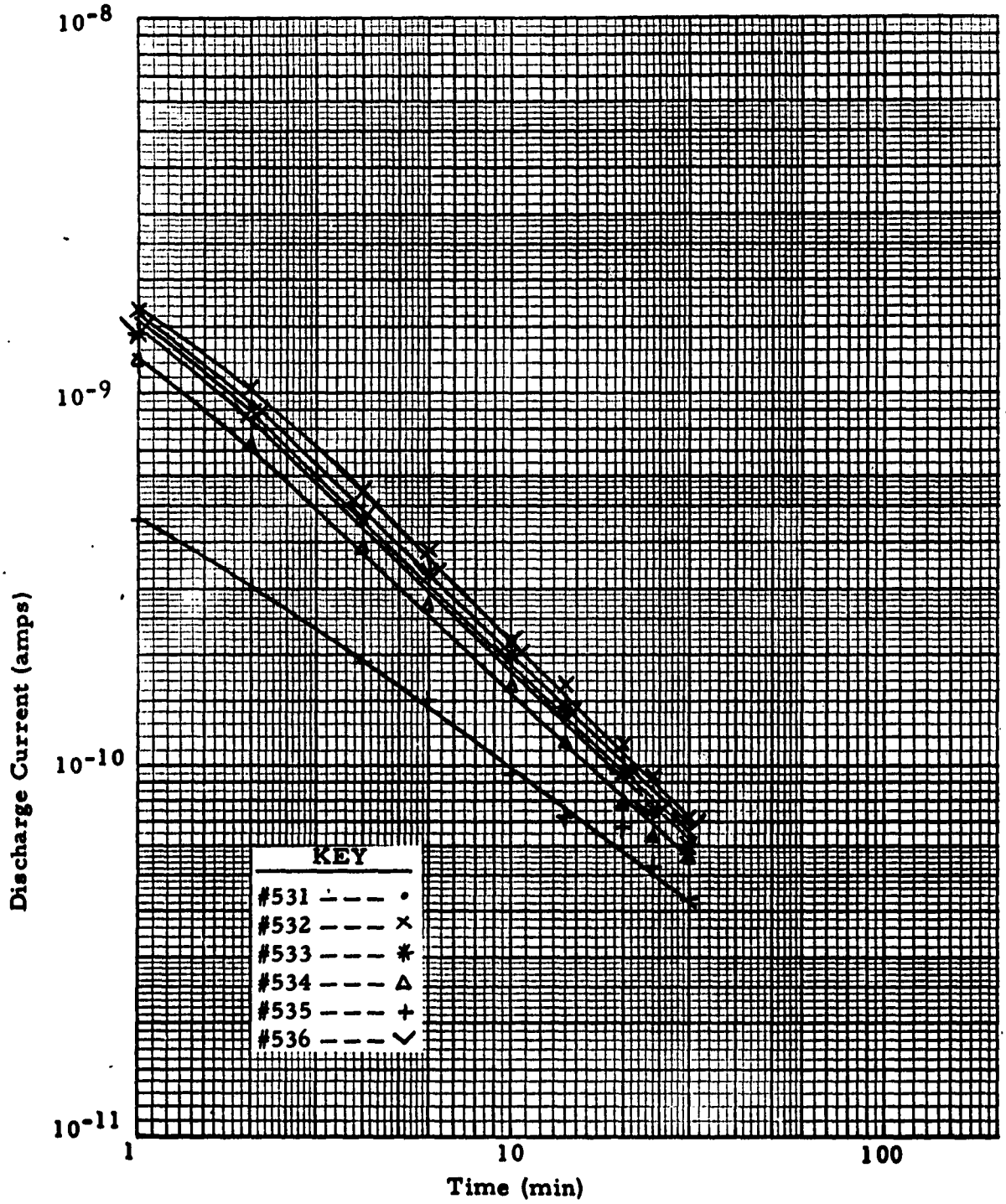
VOLTAGE-TEMPERATURE MATRIX

Voltage \ Temperature	25°C	85°C	125°C	150°C
25 V			100	
50 V	100	100	100	100
100 V	100	100	300	100
200 V	100	100	100	100



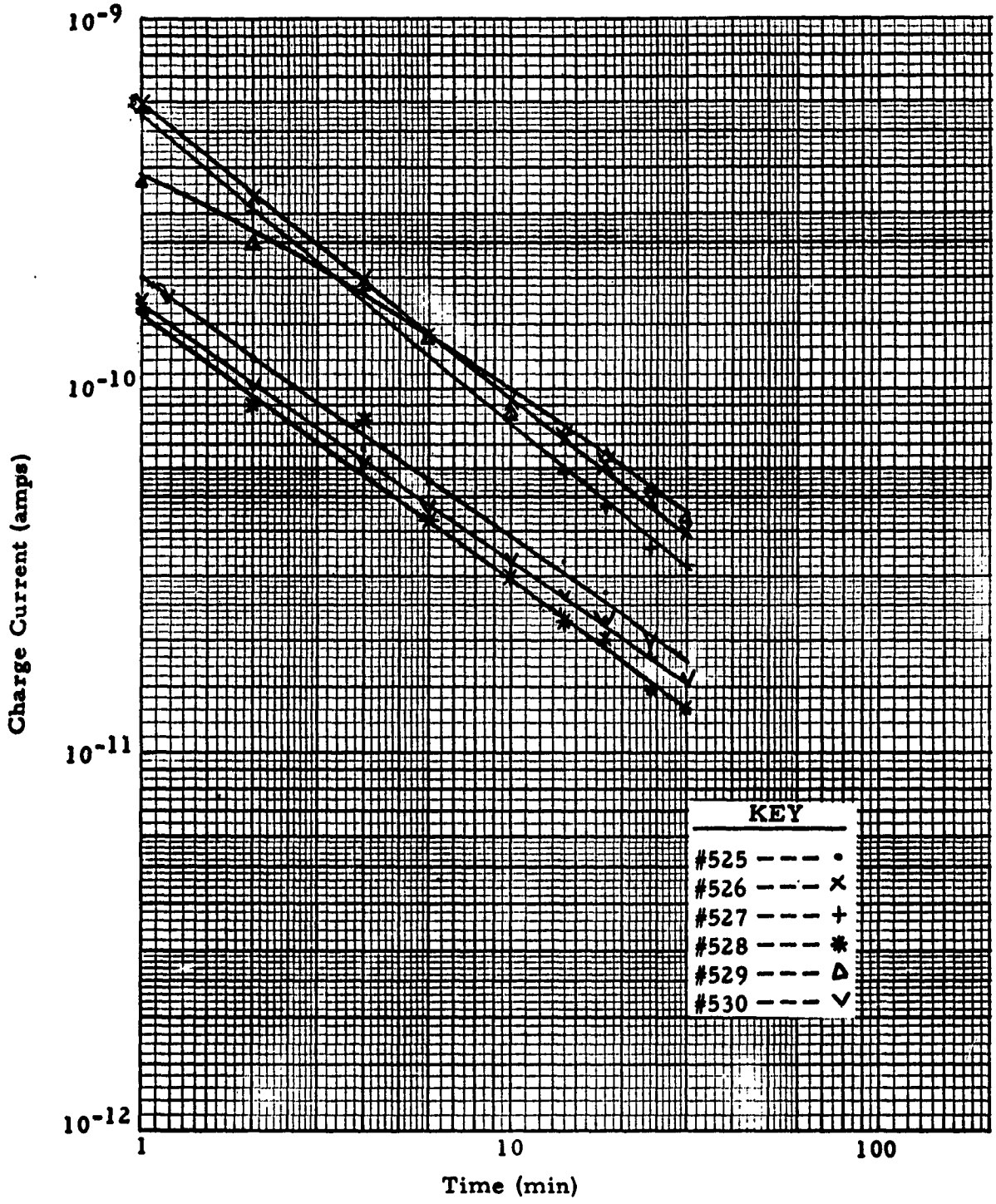
CHARGE CURRENT VS TIME AT 195 VDC, 150°C
FOR IMPROVED 0.01 µf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 6S9205)

Figure 5



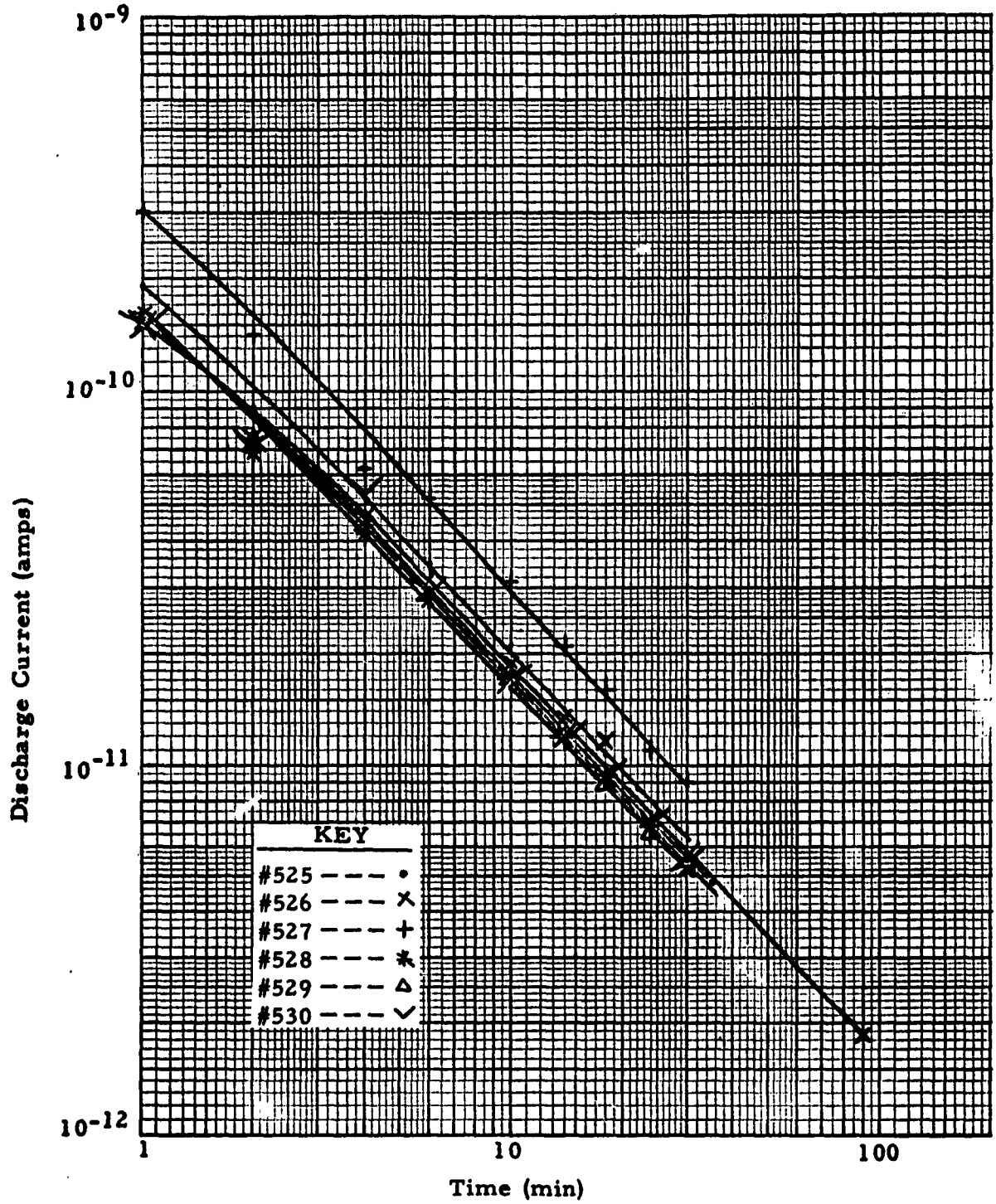
DISCHARGE CURRENT VS TIME AT 150°C
FOR IMPROVED 0.01 µf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 6S9205)
(Measured after 30 min charging with 195 VDC)

Figure 6



CHARGE CURRENT VS TIME AT 195 VDC, 25°C
FOR IMPROVED 0.01 µf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 6S9205)

Figure 7



DISCHARGE CURRENT VS TIME AT 25°C
FOR IMPROVED 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 689205)

(Measured after 30 min charging with 195 VDC)

Figure 8

Figure 9 compares current charging curves on capacitors from Lot No. 6S9205 and from a lot studied earlier. The evident features of the new lot are the greater dielectric absorption and the much lower value of steady-state charging current.

Figure 10 compares resistances of capacitors from three lots for an extended period of time at 150°C and 200 V. It seems probable that Lot No. 6S9205 does not offer the same prospect of longevity as the earlier lot of the improved version, but it appears far superior to the obsolete version. Figure 11 presents resistance with time at 150°C and 195 V for several capacitors from Lot No. 6S9205.

From the foregoing data, it is clear that the screening technique described earlier in this report cannot be applied to Lot No. 6S9205 capacitors unless the technique or conditions are modified. In particular, the condition that resistance not change more than 20% during burn-in for an acceptable capacitor must be relaxed considerably.

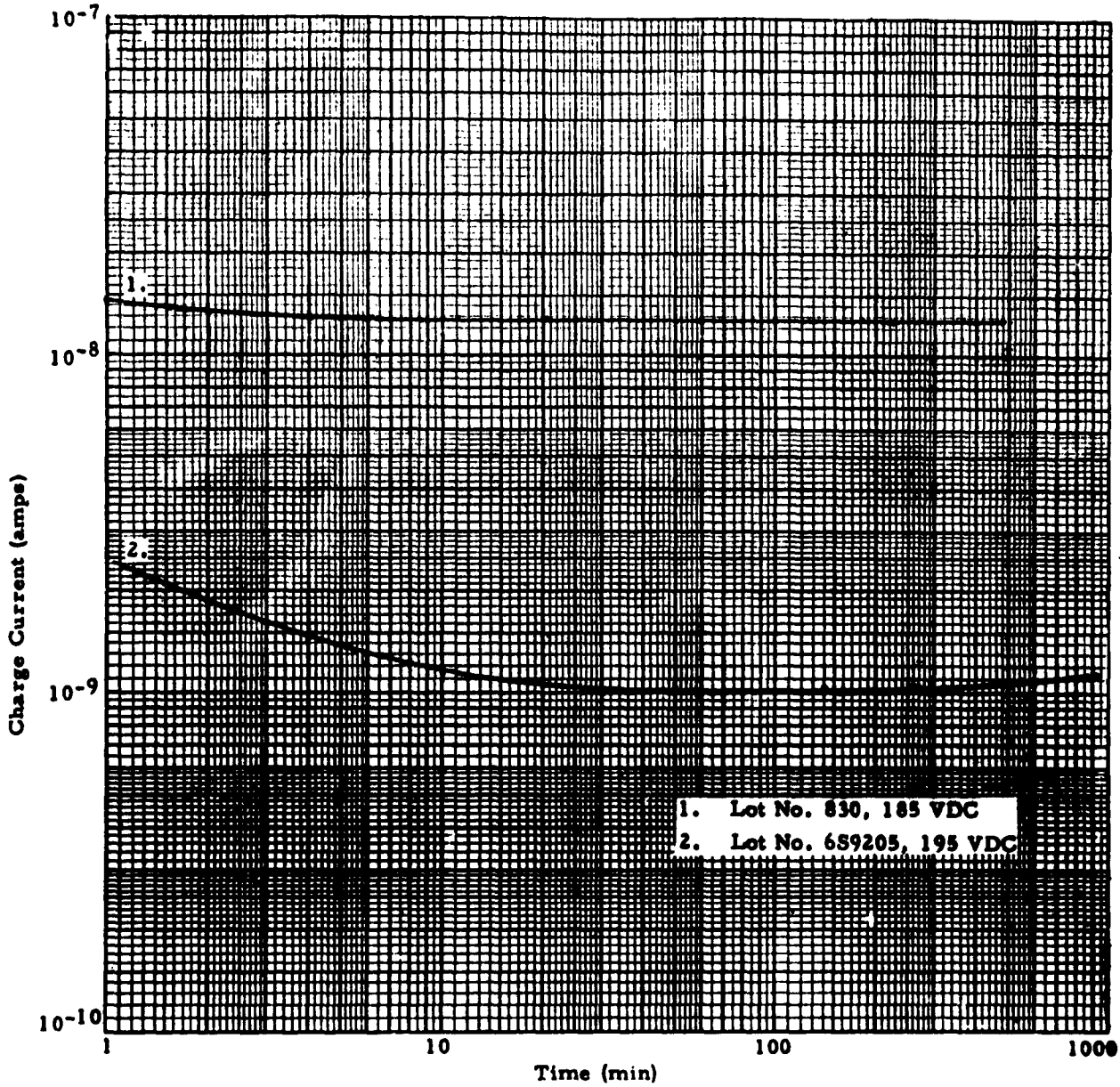
An estimate of performance for the majority of the capacitors shown in Figure 11 at other conditions of voltage and temperature may be made using relationships presented in the Eighth Quarterly Progress Report and which are combined into a single expression below:

$$t_1 = t_2 \left[\left(\frac{E_2}{E_1} \right)^n \right] \exp \left[\frac{W}{kT_1} - \frac{W}{kT_2} \right]$$

where,

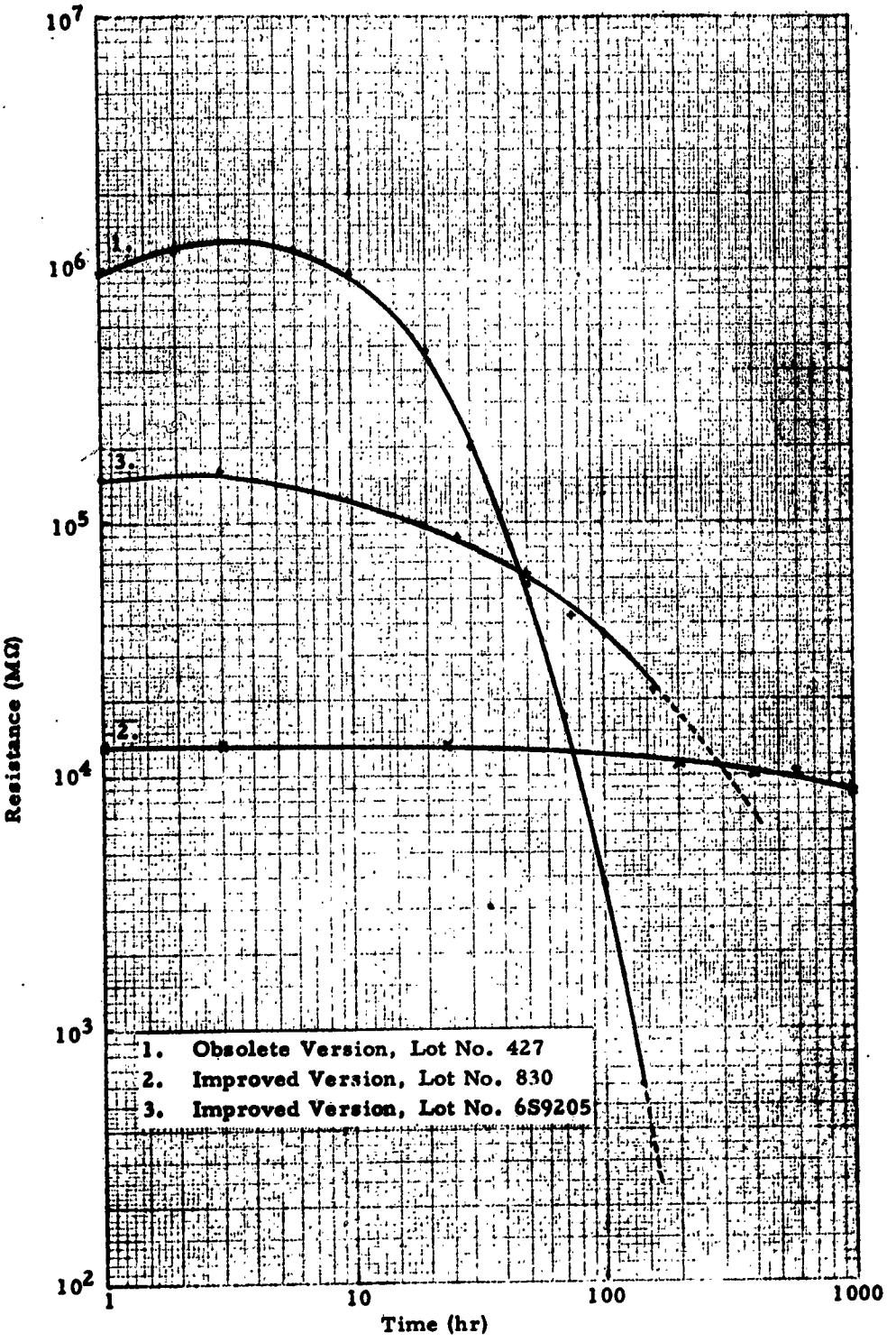
t_1 = performance time to be calculated
t_2 = 160 hr
E_2 = 195 V
E_1 = test voltage
n = 2.7
W = 0.90 eV
k = Boltzmann Constant (0.0000862 eV/°K)
T_2 = 423°K (150°C)
T_1 = test temperature

The following performances are to be considered equivalent as calculated from the above expression.



CHARGE CURRENT VS TIME AT 150°C
FOR IMPROVED 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS

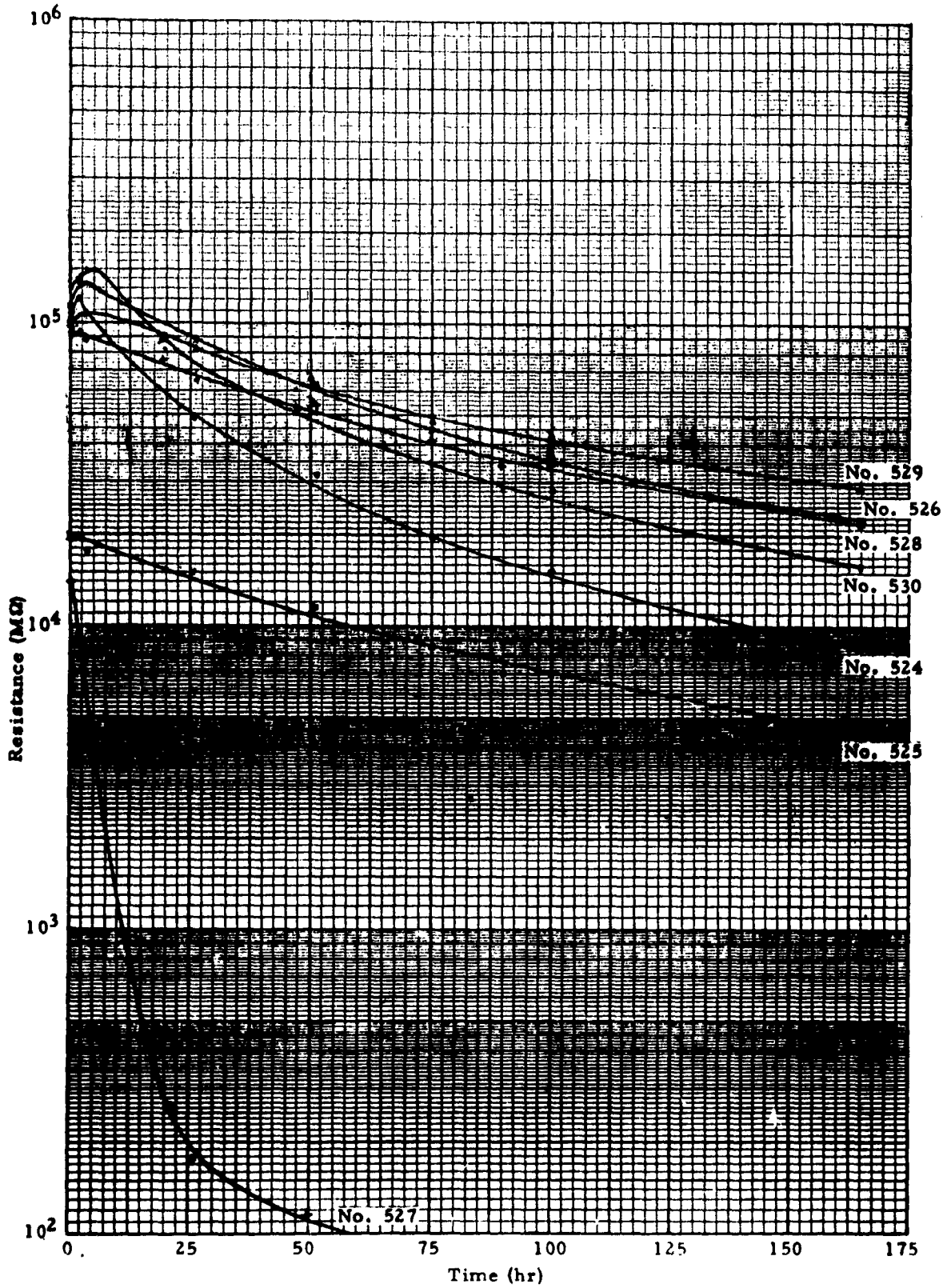
Figure 9



TYPICAL RESISTANCE VS TIME
FOR 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS

Conditions: 80 VDC/mil, 150°C

Figure 10



RESISTANCE VS TIME AT 195 VDC, 150°C
FOR 0.01 μf CASE SIZE I C67 MONOLYTHIC CAPACITORS (Lot 6S9205)

Figure 11

Equivalent Performances		
Temperature	Voltage	Time
150°C	195 V	160 hr
125°C	100 V	4,560 hr
125°C	50 V	29,700 hr
85°C	200 V	13,200 hr
85°C	100 V	86,000 hr

Examination of Lot No. 6S9205 capacitors will be continued to determine the criteria to be used for selecting potentially long-life capacitors.

4.4 Capacitors Having 1.5 Mil Dielectric Layers

The 0.022 μf C67 Case Size I MONOLYTHIC capacitors having 1.5 mil dielectric layers have not yet been manufactured.

SECTION 5

CONCLUSIONS

(1) The testing of 124 of the improved version of the 0.01 μf C67 Case Size I MONOLYTHIC capacitor under the new test conditions of 125°C and 400 VDC has now gone 4000 hr. Two of the 53 capacitors which passed both parts of the screening program are showing marked degradation.

(2) Lot No. 6S9205, consisting of 1800 capacitors (0.01 μf) of the improved version, was received during this period. Examination of these capacitors, on a sample basis, showed greater dielectric absorption and a much lower value of steady-state charging current. These preliminary tests also indicated that this lot cannot be subjected to the two-part screening program unless the screening technique or conditions are modified.

(3) Manufacture of 0.022 μf C67 Case Size I capacitors with dielectric layers 1.5 mils thick has not yet been completed.

SECTION 6

PROGRAM FOR NEXT QUARTER

(1) Examination of the 1800 C67 Case Size I MONOLYTHIC capacitors will be completed to determine criteria to be used for selecting potential long-life capacitors for voltage-temperature matrix testing.

(2) Voltage-temperature matrix testing will begin.

SECTION 7

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13. ABSTRACT

Testing of C67 Case Size I Monolythic capacitors of the improved version continues to demonstrate the long-life capability of this capacitor at use conditions. The testing of 124 of these capacitors at 200 VDC and 125°C for 2500 hours without catastrophic failure, then at 400 VDC and 125°C for 4000 hours with only five displaying any degradation, continues to indicate the intrinsic high reliability of this unit.

Some properties of 1800 capacitors (0.01 μf rating) prepared for the voltage temperature matrix testing were examined.

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14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.